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IS 13467 (1992): Chlorinated rubber for paints [CHD 20: Paints, Varnishes and Related Products]

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Indian Standard

CHLORINATED RUBBER FOR PAINTS — SPECIFICATION

UDC 678·473·3 : 667·6

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 3

Raw Materials for Paint Industry Sectional Committee, CHD 021

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

Chlorinated rubber is used principally in protective paints and coating where resistance to chemicals, corrosive atmosphere, presence of a large amount of moisture, daily decontamination of surface by washing and even continuous immersion is required.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CHLORINATED RUBBER FOR PAINTS — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of test for the material commercially known as chlorinated rubber for paints.

2 REFERENCES

The Indian Standards listed below are the necessary adjuncts to this standard:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
354 (Part 1) : 1987	Methods of sampling and test for resins for paints : Part 1 General test methods (*second revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
2263 : 1979	Method of preparation of indicator solutions for volumetric analysis (*first revision*)
2316 : 1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis (*second revision*)
2630 : 1973	Nitrobenzene (*first revision*)
4542 : 1983	Colorimetric methods for determination of iron
4905 : 1968	Methods for random sampling

3 GRADES

3.1 The material shall be of the following three grades depending upon its viscosity:

a) Grade 10,
b) Grade 20, and
c) Grade 40.

3.1.1 The grade number is the nominal viscosity of 20 percent solution in centipoise of the grade.

4 REQUIREMENTS

4.1 Description

The material shall be white to off white colour powder and free from foreign matter.

4.2 The material shall also comply with the requirements given in Table 1.

5 KEEPING PROPERTY

The material, when stored in normal conditions for 12 months, shall retain its original properties and conform to requirements given in **4**.

6 PACKING AND MARKING

6.1 Packing

The material shall be suitably packed as agreed to between the purchaser and the manufacturer.

6.2 Marking

Each container shall be marked with the following information:

a) Name of the material,
b) Grade,
c) Indication of source of the manufacture,
d) Net mass of the material,
e) Batch or lot number in code or otherwise, and
f) Month and year of manufacture.

7 SAMPLING

Representative samples shall be drawn as prescribed in Annex B.

Table 1 Requirements for Chlorinated Rubber for Paints

(*Clause* 4.2)

Sl No.	Characteristic	Requirement for Chlorinated Rubber			Method of Test, Ref to Annex A
		Gr10	Gr20	Gr40	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Volatile matter, percent by mass, 60°C, 1 h under vacuum, *Max*	2·0	2·0	2·0	A-2
ii)	Viscosity*				A-4
	a) 20 percent solution (m/m), centipoise	10±2	20±2	40±4	A-4.1
	b) 30 percent solution (m/m), sec	45±5	70±5	140±10	A-4.2
iii)	Clarity of the film	Shall be clear and free from bits			A-5
iv)	Colour, *Max*	12	12	12	A-6
v)	Chlorine, percent by mass, *Min*	60·0	60·0	60·0	A-7
vi)	Free chlorine,	— To pass the test —			A-8
vii)	Ash, percent by mass, *Max*	0·50	0·50	0·50	A-9
viii)	Acidity (as HCl), percent by mass, *Max*	0·05	0·05	0·05	A-10
ix)	Iron content, ppm, *Max*	10	10	10	A-11

* Either (a) or (b) method can be used. However, in case of dispute, method (a) would be the referee method.

ANNEX A

(*Table* 1)

REQUIREMENTS FOR CHLORINATED RUBBER FOR PAINTS

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 VOLATILES, PERCENT BY MASS

A-2.1 Apparatus

A-2.1.1 *Petri-dish*

Approximately 7 to 8 cm diameter and 2 cm deep.

A-2.1.2 *Vacuum Oven*

Provided with temperature indicator and control, vacuum regulating device and capable of maintaining recommended temperature and vacuum.

A-2.1.3 *Vacuum Pump*

A-2.2 Procedure

Weigh accurately about 1 to 1·5 g of the material and spread uniformly in a petri-dish. Put the petri-dish in vacuum oven maintained at 60°C and a pressure of about 133·322 Pa. After one hour remove the petri-dish and keep in the desiccator. Allow it to cool to room temperature and reweigh. Calculate loss of mass.

NOTE — $1 \text{ torr} = \frac{1}{760} \text{ atm} = 13\,332{\cdot}2 \text{ Pa}$

A-2.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = initial mass of the sample, and

M_2 = final mass of the sample.

A-3 PREPARATION OF SOLUTION OF CHLORINATED RUBBER IN O-XYLENE

A-3.1 Reagent

A-3.1.1 *O-xylene*

A-3.2 Procedure

Prepare 30 percent *m/m* solution of chlorinated rubber in o-xylene by weighing accurately 60·0 g of the sample in 500-ml beaker and adding 140·0 g of o-xylene in it. Stir with glass rod. Note the total mass of the beaker and warm on water bath or hot plate. Stir well till whole mass dissolves in the xylene. Solution should be clear. Reweigh the beaker and compensate for loss of solvent. Store the solution in stoppered bottle and use it for tests prescribed in **A-4**, **A-5**, **A-6** and **A-8**.

A-4 DETERMINATION OF VISCOSITY

A-4.1 Ostwald u-Tube Viscometer Method

Determine the viscosity of 20 percent (*m/m*) solution in toluene as per the method mentioned in **8.3** of IS 354 (Part 1) : 1987 using u-tube viscometer and report the result.

A-4.2 Flow Cup (Ford Cup Viscometer No. 4)

Determine the viscosity of 30 percent (*m/m*) solution, as prepared in **A-3**, as per the method mentioned in **8.2** of IS 354 (Part 1) : 1987 using flow cup (ford cup viscometer No. 4) and report the result.

A-5 CLARITY OF THE FILM

Prepare the solution as in **A-3**. Take a draw-down of the 30 percent solution on a clean glass plate with 6 millibar applicator (150 microns). Allow to dry the film in completely dust free room for 30 minutes and observe the clarity of film.

A-6 COLOUR OF 30 PERCENT (W/W) SOLUTION

A-6.1 Prepare solution as in **A-3**. Measure the colour of the solution as per standard test method of colour of transparent liquids using Gardner colour standards (*see* **6.2** of IS 354 (Part 1) : 1987.

A-6.2 Report

Report the colour as the number of the standard, most closely matching the specimen. For most precise measurement report as either matching in standard or lighter, or darker. Thus, for example, colour between 11 and 12, the increasingly darker colour description would be 11, 11+, 12− and 12.

A-7 CHLORINE CONTENT

A-7.1 Outline of the Method

The organic matter is destroyed by hot sodium peroxide and chlorine is converted to chloride. The chloride is precipitated as silver chloride with excess of standard silver nitrate, and excess of silver nitrate is determined by titration with standard thiocyanate solution using ferric alum as indicator. Nitrobenzene is added to depress the solubility of silver chloride. The amount of silver nitrate required to precipitate the chloride is the measure of total chlorine content of the sample.

A-7.2 Apparatus

Peroxide bomb similar to Parr series 2100, 22 ml gas fired.

A-7.3 Reagents and Materials

A-7.3.1 *Sodium Peroxide*

A-7.3.2 *Nitric Acid, see* IS 264 : 1976.

A-7.3.3 *Silver Nitrate Solution,* approximately 0·1 N.

A-7.3.4 *Standard Potassium Thiocyanate Solution,* 0·1 N (*see* IS 2316 : 1990).

A-7.3.5 *Ferric Ammonium Sulphate Indicator Solution,* 2 percent (*see* IS 2263 : 1979).

A-7.3.6 *Nitrobenzene*

A-7.4 Standardization of Potassium Thiocyanate Solution

A-7.4.1 *Procedure*

Weigh accurately up to 0·001 g, 0·1 to 0·2 g of silver nitrate in a conical flask and add 5 ml of 6 N nitric acid and about 25 to 30 ml of distilled water. To this add 5 ml of ferric alum indicator. Weigh accurately 10·5 g of potassium thiocyanate and dissolve it in 1 litre water in volumetric flask. Take thiocyanate solution in burette and titrate against silver nitrate solution in conical flask. At first a white precipitate is produced, rendering the liquid of milky appearance and as each drop of thiocyanate falls in, it produces a reddish brown cloud which quickly disappears on shaking. As the end point approaches the precipitate becomes flocculent and settles down. Finally one drop of thiocyanate solution produces a colour, which no longer disappears on shaking. This is the end point.

A-7.4.2 *Calculation*

$$\text{Normality of potassium thiocyanate solution} = \frac{M \times 1\,000}{169{\cdot}87 \times v}$$

where

M = mass of silver nitrate taken, and

v = volume of thiocyanate used.

A-7.5 Procedure

To peroxide bomb assembly add 0·200 to 0·225 g of finely divided sample and 15 g of Na_2O_2. Close the bomb tightly with lid and the retaining rings. Tap the bomb to dislodge the particles adhering to the cap. Set the bomb over the hole in a steel housing supported on a tripod stand. Heat the bomb with Bunsen flame gently until red ring appears on the side of the cup. After the vigorous reaction has taken place, quench the bomb in cold water. After cooling remove it from quenching bath rinse off with water and open. Place the bomb in 600-ml beaker and wash off all content in the cup, collect all washing carefully. Remove the bomb cup and rinse thoroughly with water collecting all rinsings in the same beaker. Transfer quantitatively to 500-ml glass stoppered flask add 2-3 drops of phenolphthalein indicator, neutralize with diluted nitric acid taking care to avoid loss by spattering and add little excess of dilute nitric acid. Add 2 ml of nitrobenzene and 50·0 ml of approximately 0·1 N silver nitrate solution, stopper and shake until the precipitated silver nitrate becomes a spongy mass. Add 10 to 15 ml of ferric alum indicator solution and titrate with 0·1 N standard potassium thiocyanate solution to the first pink end point in the supernatant liquid. Run a blank determination.

A-7.5.1 *Calculation*

Calculate the chlorine content as follows:

$$\text{Chlorine content, percent by mass} = \frac{(V_1 - V_2) \times N \times 3{\cdot}546}{M}$$

where

V_1 = volume of 0·1 N thiocyanate required for blank titration, in ml;

V_2 = volume of 0·1 N thiocyanate required for sample, in ml;

N = normality of thiocyanate; and

M = mass of sample taken.

A-8 FREE CHLORINE CONTENT

A-8.1 Reagent

A-8.1.1 *Starch-Iodide Solution*

Prepare starch-iodide solution by dissolving 5·0 g potassium iodide in 100 ml of water containing 10 ml of 0·2 percent starch solution.

A-8.2 Procedure

Transfer 3 to 5 ml of 30 percent *m/m* solution in o-xylene to a test tube (*see* **A-3**), add 2 to 3 ml of starch iodide solution and shake vigorously for 1 minute.

A-8.3 Observation

No blue colouration shall be observed in the aqueous layer.

A-9 ASH CONTENT

A-9.1 Apparatus

A-9.1.1 *Silica Crucible* — 50 ml capacity.

A-9.1.2 *Muffle Furnace*

Capable of maintaining temperature up to 750 ± 20°C with temperature indicator and control.

A-9.2 Procedure

Ignite a silica crucible, cool in desiccator and weigh to the nearest 0·1 mg. Weigh accurately about 15-20 g of the sample in the silica crucible. Place it on a tripod stand and heat gently with a Bunsen burner until the sample catches flame. Remove the burner and allow the sample to burn completely. Reheat from time to time to bring about complete combustion. Carry out this heating in hood having good exhaust system. Remove the crucible and place it in a furnace adjusted at a temperature 750 ± 20°C until free from carbon. When ashing is complete, cool the crucible in a desiccator and then weigh it to the nearest 0·1 mg. Continue with the ignition until constant mass is obtained.

A-9.3 Calculation

Calculate the ash content as follows:

$$\text{Ash content, percent by mass} = \frac{M_1 - M}{M_2} \times 100$$

where

M_1 = mass of crucible and ash,

M = mass of the empty crucible, and

M_2 = mass of the sample.

A-10 ACIDITY

A-10.1 Outline of the Method

Inorganic acids are allowed to dissolve in water and estimated by titration against standard alkali.

A-10.2 Reagents

A-10.2.1 *Standard Sodium Hydroxide Solution*, 0·1 N (*see* IS 2263 : 1979).

A-10.2.2 *Phenolphthalein Indicator*

Three percent solution in 50 percent aqueous ethanol (*see* IS 2630 : 1973).

A-10.3 Procedure

Place 10-15 g of the sample weighed to the nearest 0·05 g in a 250-ml flask with 100-ml of boiled and cooled distilled water. Allow it to soak for 1 hour at room temperature swirling at 10 minutes intervals. Filter the contents of the flask in 250-ml standard volumetric flask through filter paper No. 41. Give washings and dilute up to the mark. Pipette out 50-ml of diluted solution in Erlenmeyer flask. Add 2 drops of phenolphthalein indicator and titrate to a faint pink end point with 0·1 N standard sodium hydroxide solution.

A-10.4 Calculation

$$\text{Acidity (as HCl), percent by mass} = \frac{V \times N \times 18{\cdot}25}{M}$$

where

V = Volume of 0·1 N NaOH required for titration, in ml;

N = normality of NaOH solution; and

M = weight of the sample taken.

A-11 IRON CONTENT (COLORIMETRIC METHOD)

Convert 15 to 20 g of sample into ash by ash content method (*see* **A-9**). Dissolve this ash in concentrated hydrochloric acid and dilute the solution to appropriate volume and determine the iron content by colorimetric method as prescribed in IS 4542 : 1983.

ANNEX B

(*Clause* 7)

SAMPLING PLAN OF CHLORINATED RUBBER FOR PAINTS

B-1 LOT

The bags of chlorinate rubber of the same grade belonging to the same batch of production shall constitute a lot. Each lot shall be tested separately.

B-2 SCALE OF SAMPLING

The number of bags to be selected for sampling shall be as given in Table 2.

These bags shall be selected at random from the lot, as prescribed in IS 4905 : 1968.

B-3 PREPARATION OF TEST SAMPLES

From each selected bag, a representative portion of material shall be taken, which shall be sufficient for carrying out tests for all characteristics given in the specification.

B-3.1 Composite Sample

Out of these portions, a small but equal quantity of material shall be taken and mixed thoroughly to make a composite sample.

B-3.2 Individual Sample

The remaining portion of material from each selected bag shall constitute an individual test sample.

B-4 NUMBER OF TESTS

B-4.1 Test for **4.1** (Description) and Sl No. (v) of Table 1 (Chlorine content) shall be performed on the individual sample (**B-3.2**). The tests for all other characteristics of **4.2** shall be conducted on the composite sample (**B-3.1**).

B-5 CRITERIA FOR CONFORMITY

The lot shall be deemed to conform to the specification if all the test results according to **B-4.1** satisfy the corresponding requirements.

Table 2 Scale of Sampling

(*Clause* B-2)

Number of Bags in the Lot N	Number of Bags to be Selected n
(1)	(2)
Up to 50	3
51 to 100	4
101 to 200	5
201 to 300	6
301 and above	7

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